U-tube method, IBR approved for §1065.705(c).

(13) ISO 14596:2007, Petroleum products—Determination of sulfur content—Wavelength-dispersive X-ray fluorescence spectrometry, IBR approved for §1065.705(c).

(14) ISO 14597:1997, Petroleum products—Determination of vanadium and nickel content—Wavelength dispersive X-ray fluorescence spectrometry, IBR approved for §1065.705(c).

(15) ISO 14644–1:1999, Cleanrooms and associated controlled environments, IBR approved for §1065.190(b).

(f) *NIST material.* The following documents are available from National Institute of Standards and Technology, 100 Bureau Drive, Stop 1070, Gaithersburg, MD 20899–1070, (301) 975–6478, or *www.nist.gov*:

(1) NIST Special Publication 811, 2008 Edition, Guide for the Use of the International System of Units (SI), March 2008, IBR approved for §§1065.20(a) and 1065.1005.

(2) NIST Technical Note 1297, 1994 Edition, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, IBR approved for §1065.1001.

(g) *SAE International material.* The following standards are available from SAE International, 400 Commonwealth Dr., Warrendale, PA 15096–0001, (724) 776–4841, or *http://www.sae.org*:

(1) SAE 770141, 1977, Optimization of Flame Ionization Detector for Determination of Hydrocarbon in Diluted Automotive Exhausts, Glenn D. Reschke, IBR approved for §1065.360(c).

(2) SAE J1151, Methane Measurement Using Gas Chromatography, stabilized September 2011, IBR approved for §§1065.267(b) and 1065.750(a).

[79 FR 23818, Apr. 28, 2014]

Subpart L—Methods for Unregulated and Special Pollutants

SOURCE: 79 FR 23820, Apr. 28, 2014, unless otherwise noted.

§ 1065.1101 Applicability.

This subpart specifies procedures that may be used to measure emission constituents that are not measured (or not separately measured) by the test procedures in the other subparts of this part. These procedures are included to facilitate consistent measurement of unregulated pollutants for purposes other than compliance with emission standards. Unless otherwise specified in the standard-setting part, use of these procedures is optional and does not replace any requirements in the rest of this part.

SEMI-VOLATILE ORGANIC COMPOUNDS

§ 1065.1103 General provisions for SVOC measurement.

The provisions of §§1065.1103 through 1065.1111 specify procedures for measuring semi-volatile organic compounds (SVOC) along with PM. These sections specify how to collect a sample of the SVOCs during exhaust emission testing, as well as how to use wet chemistry techniques to extract SVOCs from the sample media for analysis. Note that the precise method you use will depend on the category of SVOCs being measured. For example, the method used to measure polynuclear aromatic hydrocarbons (PAHs) will differently from the method used to measure dioxins. Follow standard analytic chemistry methods for any aspects of the analysis that are not specified.

(a) Laboratory cleanliness is especially important throughout SVOC testing. Thoroughly clean all sampling system components and glassware before testing to avoid sample contamination. For the purposes of this subpart, the sampling system is defined as sample pathway from the sample probe inlet to the downstream most point where the sample is captured (in this case the condensate trap).

(b) We recommend that media blanks be analyzed for each batch of sample media (sorbent, filters, etc.) prepared for testing. Blank sorbent modules (i.e., field blanks) should be stored in a sealed environment and should periodically accompany the test sampling system throughout the course of a test, including sampling system and sorbent module disassembly, sample packaging, and storage. Use good engineering judgment to determine the frequency with which you should generate field blanks. The field blank sample

should be close to the sampler during testing.

(c) We recommend the use of isotope dilution techniques, including the use of isotopically labeled surrogate, internal, alternate, and injection standards.

(d) If your target analytes degrade when exposed to ultraviolet radiation, such as nitropolynuclear aromatic hydrocarbons (nPAHs), perform these procedures in the dark or with ultraviolet filters installed over the lights.

(e) The following definitions and abbreviations apply for SVOC measurements:

(1) *Soxhlet extraction* means the extraction method invented by Franz von Soxhlet, in which the sample is placed in a thimble and rinsed repeatedly with a recycle of the extraction solvent.

(2) *XAD–2* means a hydrophobic crosslinked polystyrene copolymer resin adsorbent known commercially as Amberlite® XAD®-2, or an equivalent adsorbent like XAD–4.

(3) *Semi-volatile organic compound (SVOC)* means an organic compound that is sufficiently volatile to exist in vapor form in engine exhaust, but that readily condenses to liquid or solid form under atmospheric conditions. Most SVOCs have at least 14 carbon atoms per molecule or they have a boiling point between (240 and 400) °C. SVOCs include dioxin, quinone, and nitro-PAH compounds. They may be a natural byproduct of combustion or they may be created post-combustion. Note that SVOCs may be included in measured values of hydrocarbons and/or PM using the procedures specified in this part.

(4) *Kuderna-Danish concentrator* means laboratory glassware known by this name that consists of an air-cooled condenser on top of an extraction bulb.

(5) *Dean-Stark trap* means laboratory glassware known by this name that uses a reflux condenser to collect water from samples extracted under reflux.

(6) *PUF* means polyurethane foam.

(7) *Isotopically labeled* means relating to a compound in which either all the hydrogen atoms are replaced with the atomic isotope hydrogen-2 (deuterium) or one of the carbon atoms at a defined position in the molecule is replaced with the atomic isotope carbon-13.

§ 1065.1105   Sampling system design.

(a) *General.* We recommend that you design your SVOC batch sampler to extract sample from undiluted emissions to maximize the sampled SVOC quantity. To the extent practical, adjust sampling times based on the emission rate of target analytes from the engine to obtain analyte concentrations above the detection limit. In some instances you may need to run repeat test cycles without replacing the sample media or disassembling the batch sampler.

(b) *Sample probe, transfer lines, and sample media holder design and construction.* The sampling system should consist of a sample probe, transfer line, PM filter holder, cooling coil, sorbent module, and condensate trap. Construct sample probes, transfer lines, and sample media holders that have inside surfaces of nickel, titanium or another nonreactive material capable of withstanding raw exhaust gas temperatures. Seal all joints in the hot zone of the system with gaskets made of nonreactive material similar to that of the sampling system components. You may use teflon gaskets in the cold zone. We recommend locating all components as close to probes as practical to shorten sampling system length and minimize the surface exposed to engine exhaust.

(c) *Sample system configuration.* This paragraph (c) specifies the components necessary to collect SVOC samples, along with our recommended design parameters. Where you do not follow our recommendations, use good engineering judgment to design your sampling system so it does not result in loss of SVOC during sampling. The sampling system should contain the following components in series in the order listed:

(1) Use a sample probe similar to the PM sample probe specified in subpart B of this part.

(2) Use a PM filter holder similar to the holder specified in subpart B of this part, although you will likely need to use a larger size to accommodate the high sample flow rates. We recommend using a 110 mm filter for testing spark ignition engines or engines that utilize exhaust aftertreatment for PM removal and a 293 mm filter for other engines. If you are not analyzing separately for SVOCs in gas and particle